

Patent
Attorney's Docket No. 025219-317

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)
Patrick Hourquebie et al.) Group Art Unit: 1714
Application No.: 09/786,985) Examiner: Tae H. Yoon
Filed: May 21, 2001) Confirmation No.: 8711
For: Material with Improved Resistance to)
Thermal Ageing and its Method of)
Production)

DECLARATION UNDER 37 C.F.R. §1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, Patrick Hourquebie, hereby state as follows:

EDUCATION AND BACKGROUND

1. I am an inventor in the above-identified application. I hold an Engineering Diploma from École Nationale Supérieure de Chimie et de Physique de Bordeaux and a Ph.D from the University of Bordeaux I. A list of publications with which I was involved is attached as Exhibit 1. I am currently head of the Organic and Dielectric Materials Laboratory at CEA/Le Ripault which specializes in the synthesis, formulation and transformation of polymers.

2. I have reviewed and am familiar with U.S. Patent No. 5,254,633, issued to Han et al., and PCT Publication WO 96/21694, inventors Conn et al.

Application Serial No. 09/786,985

Atty. Docket No. 025219-317

CONDUCTIVITY

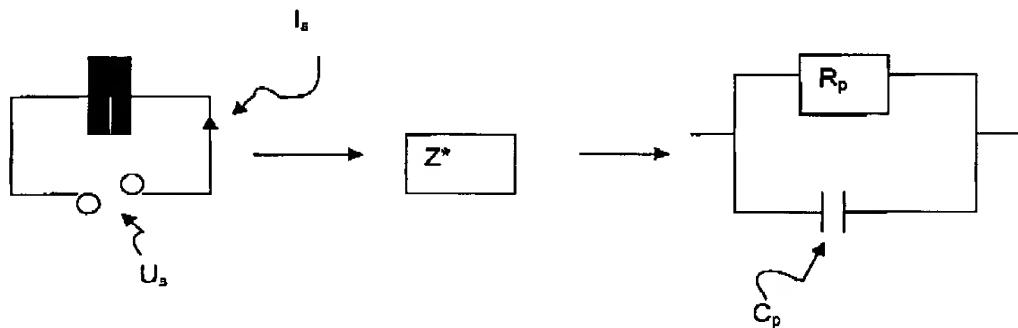
3. The material defined by the pending claims of the present '985 application is an insulating material having improved resistance to thermal ageing. These materials contain 10 to 5000 ppm of a conducting polymer dispersed in or on an insulating polymer and show no heterogeneity on a scale of $0.1 \mu\text{m}$ as observed under scanning electron microscopy. The materials as prepared by the methods defined in the pending claims do not lead to an increase in conductivity of the insulating polymer used.

4. The fact that the conductivity of the prepared material is not increased with the steps and concentrations of the process of the invention as claimed in the present application cannot be overlooked in the use of the material as an insulator for very high voltage cable (see example 4 of the present application).

5. The lack of increase in conductivity of the insulating polymer can be shown by measuring the dielectric losses of the materials. Any increase in the electrical conductivity of a material is shown by an increase in dielectric losses.

6. The dielectric losses of materials according to the pending claims were tested by measuring dielectric losses according to frequency carried out in the conventional way by measuring the complex impedance of the material between two electrodes using an impedance meter. Figure 1 illustrates the principle of measuring complex impedance:

Figure 1: Diagram of the principle of measuring complex impedance.



Application Serial No. 09/786,985

Atty. Docket No. 025219-317

The sample represents a capacity C_p in parallel with a resistor R_p .

$$\text{Therefore: } \frac{1}{Z^*} = \frac{1}{R_p} + i\omega C_p$$

$$\text{With } \epsilon^1 = \frac{C_p}{C_0} \text{ and } \epsilon^{11} = \frac{1}{R_p C_0 \omega}$$

C_p being the vacuum capacity of the capacitor.

$\omega = 2\pi f$ with f the measuring frequency.

The loss angle can be calculated for the defined material by $\tan \delta = \frac{\epsilon^{11}}{\epsilon^1}$

7. The measurement of dielectric losses on thin films of insulating polymer in alloy form according to the materials as defined in the pending claims, varying from 1000 to 4000 ppm shows no increase in dielectric losses, as shown in Figure 2. The lack of increase in dielectric losses proves that the conductivity of the insulating polymer does not change.

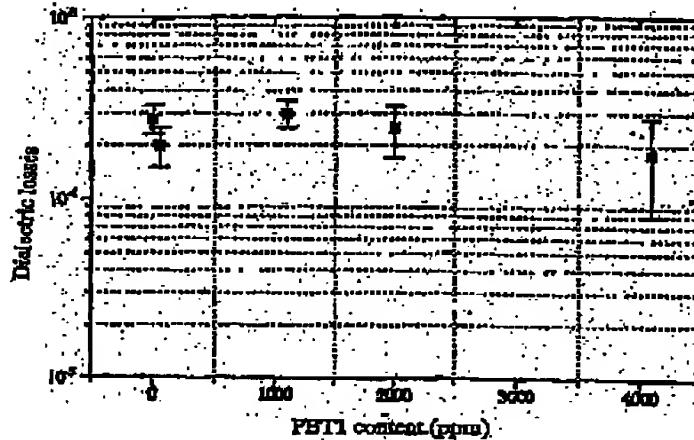


Figure 2: Changes in dielectric losses in a polyethylene and poly (butyl-3 thiophene) mixture at 10 kHz.

NO HETEROGENEITY

8. The heterogeneity defined in the claims would be understood by one of skill in the art. The homogeneity of a material must be assessed by starting from the lowest magnification and increasing magnification gradually to find at what point the heterogeneity occurs. Any method that allows for observing a contrast between the two

Application Serial No. 09/786,985

Atty. Docket No. 025219-317

materials can be used. This is the case with SEM, which allows for observing a contrast due to the difference in conductivity of the two materials making up the mixture. If a very strong magnification is directly used, the risk is run of observing an image that seems to be homogeneous, but which is only the image of one of the two constituents. However, a skilled person who tries to define the homogeneity of the mixture will observe the materials at increasing magnification as described. Thus, one of skill in the art would be able to use the claimed invention from the information provided in the claims.

9. The materials defined in the pending claims are homogeneous to the point that the structure obtained is a molecular alloy where all the conjugated polymer/conjugated polymer interactions have been replaced by conjugated polymer/insulating polymer interactions. The quality of mixing used in the process as claimed results in no heterogeneity on a scale of 0.1 μ m as claimed and allows for the increased lifetime of the material.

10. Further, more detailed measurement of the homogeneity of the prepared materials than those obtained with SEM by using AFM techniques have since been made. They have not allowed a distinction between the two polymers in the materials.

11. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name: _____
Patrick Hourquebie

Date: _____

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BURNS DOANE

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Exhibit 1

List of publications:

"Trapping and detrapping of electrical charges in polymers: How to reach materials with improved dielectric strength?"

P. HOURQUEBIE, J. BIGARRÉ, Ph. MAZABRAUD, H. JANAH
(Written and oral paper at CSC4 Tours, July 2001)

"The space charge in new insulating materials for very high voltage DC cable".

J. MATALLANA, J. BIGARRÉ, S. AGNEL, P. HOURQUEBIE, A. TOUREILLE, H. JANAH, P. MIREBEAU, B. ALADENIZE, R. COELHO. (Written and oral paper at CSC4 Tours, July 2001)

"Determination of the spatial distribution of trapped charges in insulators: Application of the electrostatic mirror method to polymers".

C. ATTARD, J. BIGARRÉ, P. HOURQUEBIE (Written and oral paper at the SFE 2000 congress, Montpellier, July 2000)

"Study of space charge behaviour in new insulating materials for very high voltage DC cable application".

J. MATALLANA, J. BIGARRÉ, P. HOURQUEBIE, S. AGNEL, A. TOUREILLE, P. MIREBEAU, H. JANAH, B. ALADENIZE, R. COELHO. (Written and oral paper at the SPE 2000 congress, Montpellier, July 2000)

"Charge trapping characterization by the means of a scanning electron microscope beam: Application to film insulators".

J. Bigarré, P. Hourquebie

JOURNAL OF APPLIED PHYSICS, Vol 85, No. 9, May 1999

"Infrared switching electroemissive devices based on highly conducting polymers"

P. Topert, P. Hourquebie

Thin Solid Films, No. 352, pp 243-248, 1999

"Preparation of a soluble N and α -alkylated polyanilines using a chemical biphasic process"

A. Falcon, A. Longeon, D. Marsacq, P. Hourquebie, A. Duchêne
Synthetic Metals, Vol. 101, pp 647-648, May 1999.

"Metallic properties of polythiophene-based conducting polymers"

P. Buvat, P. Hourquebie

Synthetic Metals, Vol. 101, pp 17-18, May 1999.

"Copolymères à base d'aniline substituée par un groupement alkyle: synthèse d'ortho-alkylanilines (Aniline-based copolymers substituted by an alkyl group: synthesis of ortho-alkylanilines)"

A. Duchêne, P. Hourquebie, A. Falcon

Journal de Chimie Physique, Vol. 95, No. 6, pp 1213-1215, June 1998.

"Synthèse et propriétés infrarouge de copolymères à base d'éthylénedithiocystophène et d'octylthiophène (Synthesis and infrared properties of copolymers with ethylenedithiocystophene and octylthiophene bases)".

P. Buvat, P. Hourquebie.

Journal de Chimie Physique, Vol. 93, No. 6, pp 1005-1009, June 1998.

"Evaluation of space charge properties in polymers by using a scanning electron microscope"

D. Marsacq, P. Hourquebie, H. Janah

Le Vide: Science, technique et applications No. 287, pp 727-731, March 1998.

"Aging of polyethylene and conducting polymer blends"

Ph. Matsbrand, P. Hourquebie, H. Janah

ACS, Polymer Preprints, Vol. 39, No. 1, pp 149-150, March 98

"Microwave and optical properties of conducting polymers: From basic research to applications".

P. Hourquebie, P. Buvat, D. Marsacq

ACS, Polymer Preprints, Vol. 2, No. 2, pp 524-525, September 97

"Visible infrared properties of regioregular poly(3-alkylthiophenes)"

P. Buvat, P. Hourquebie.

Macromolecules 1997, Vol. 30, Iss. 9, pp 2685-2692

"Microwave and optical properties of soluble conducting polymers"

P. Hourquebie, B. Blondel, and S. Dhaene

Synthetic Metals, 1997, 85, pp 1437-1438.

"Modélisation des propriétés hyperfréquences des polymères conducteurs (Modelling of the hyper-frequency properties of conductor polymers)"

P. Hourquebie, L. Olmedo, J. Prost, JP Pouget.

Journal de Chimie Physique, Vol. 92, No. 4, pp 1005-1009, April 1995.

"Microwave Properties of Conductive Polymers"

L. Olmedo, P. Hourquebie, F. Jousse.

Synthetic Metals, No. 69, pp 205-208, 1995.

"Influence of structural parameters of conducting polymers on their microwave properties"

P. Hourquebie, L. Olmedo

Synthetic Metals, No. 65, pp 19-26, 1994.

"Microwave Absorbing Materials Based on Conducting Polymers"

L. Olmedo, P. Hourquebie, F. Jousse.

Advanced Materials, Vol. 5, No. 5, pp 373-377, 1993

"Microwave Properties of Conductive Polymer Composites"

P. Hourquebie, L. Olmedo, Ch. Deleuze.

Springer Series in Solid-State Sciences, Vol. 107, pp 125-129, 1992

Participation in books:

"Microwave and optical properties of conducting polymers: From basic research to applications"

P. Hourquebie, P. Buvat, D. Marsacq

ACS book, Symposium Series No. 726/Field Responsive Polymers:

Electroresponsive, Photoresponsive and Responsive Polymers in Chemistry and Biology. Chapter 1. 1999.

"Microwave properties of conducting polymers"

L. Olmedo, P. Hourquebie, F. Jousse,

in "Handbook of Organic and Conductive Molecules and Polymers".

H. S. Nalwa Ed, John Wiley, 1997, Vol. 3, pp 367-428.